

# PATENT ABSTRACTS OF JAPAN

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## (54) DRYING METHOD OF COATING FILM

### (57)Abstract:

**PROBLEM TO BE SOLVED:** To form a coating film having excellent surface smoothness by controlling the drying rate of the coating film obtained by applying a coating liquid to suppress the mottling ununiformity in a drying process for drying the coating film formed by applying the coating liquid having 0.5 to 100 Pa.s viscosity at 0.1 sec<sup>-1</sup> shear rate at 25°C to have ≥50 µm wet film thickness.

**SOLUTION:** In the drying process for drying the coating film formed by applying the coating liquid having 0.5 to 100 Pa.s viscosity at 0.1 sec<sup>-1</sup> shear at 25°C to have ≥50 µm wet film thickness, the reduction rate (D/D<sub>0</sub>)/Δt of solvent content during the interval until the coating film having 95 mass% solvent content D<sub>0</sub> just after the coating is dried when the solvent content in the coating film just after the coating is expressed by D<sub>0</sub> g/m<sup>2</sup> and the solvent content in the coating film at a certain time in the drying is expressed by D g/m<sup>2</sup> is 0.01–0.06 sec<sup>-1</sup>.

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

[Field of the Invention] This invention relates to the technique which dries while controlling especially a rate of drying about the desiccation approach of a paint film of having applied and obtained coating liquid.

**[0002]**

[Description of the Prior Art] As an approach of drying the paint film which applied and obtained coating liquid, the temperature at the time of initial desiccation of the photosensitive material which applied and obtained the non-setting system spreading constituent to JP,50-20355,A is chosen from a 30-100-degree C temperature requirement, and the method of being a 50-150-degree C temperature requirement, and setting up highly 20-50 degrees C the drying temperature at the time of anaphase desiccation rather than initial drying temperature is indicated.

**[0003]**

[Problem(s) to be Solved by the Invention] With a means to spray the heated air which is widely used as a means to dry the paint film applied on the base material, the so-called mottle nonuniformity which a paint film front face is disturbed by the sprayed air, and loses surface smoothness is produced.

Therefore, although this mottle nonuniformity can be weakened by suppressing the rate of flow of the air which reaches a paint film, if the clearance rate of the solvent from a paint film is quick, a paint film front face will be disturbed by stagnation of floating by the solvent migration in a paint film, and the solvent which came out of the paint film, and mottle nonuniformity will be produced like the point.

[0004] It is in forming the paint film front face which it succeeds in this invention in view of the above-mentioned situation, controls mottle nonuniformity when the object controls the rate of drying of the paint film which applied and obtained coating liquid, and has the outstanding smooth nature.

**[0005]**

[Means for Solving the Problem] The above-mentioned object of this invention was attained by the following configurations.

[0006] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa·s - 100 Pa·s in 0.1sec-1 at 50 micrometers or more of wet thickness at 1.25 degrees C When the solvent content in the paint film in a certain time of day under g/m (D0)2 and desiccation is made into (D) g/m<sup>2</sup> for the solvent content in the paint film immediately after spreading, The desiccation approach of the paint film characterized by the reduction rate ( $\Delta D/D_0$ ) /  $\Delta t$  of solvent content until 95 mass % of D0 dries from a paint film being 0.01-0.06sec-1.

[0007]  $\Delta t$ : The desiccation approach of the paint film given in one characterized by absolute value  $|\Delta T/\Delta t|$  of the change rate of paint film temperature until 95 mass % of the solvent in the solvent content variation aforementioned paint film in the difference of a certain time of day t1 and t2 ( $t_1 < t_2$ ) under desiccation and the paint film between  $t_2 - t_1$   $\Delta D/\Delta t$  dries from a paint film being 0.5-7 degrees C/sec.

[0008] A shear rate at the desiccation process which dries the paint film to which viscosity applied the

coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness in the amount of paint film temperature changes of 2.25 degrees C between delta T:delta t The desiccation approach of the paint film which is while ambient temperature until 95 mass % of the solvent in a paint film dries from a paint film is 20-85 degrees C, and is characterized by establishing at least 20-degree C temperature gradient, and making it go up gradually.

[0009] The desiccation approach of the paint film characterized by absolute value  $|\Delta T/\Delta t|$  of the change rate of paint film temperature until 95 mass % of the solvent in a paint film dries from a paint film being 0.5-7 degrees C/sec at the desiccation process at which a shear rate dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s at 50 micrometers or more of wet thickness in 0.1sec-1 at 3.25 degrees C.

[0010] The desiccation approach of the paint film given in three which is while ambient temperature until 95 mass % of the solvent in said paint film dries from a paint film is 20-85 degrees C, and is characterized by establishing at least 20-degree C temperature gradient, and making it go up gradually.

[0011] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness at 4.25 degrees C Desiccation ambient temperature (a) of a period with more solvent contents in a paint film than one half of the solvent contents in the paint film immediately after spreading is made below into the glass transition point (Tg) of the binder contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this glass transition point (Tg) said more than desiccation ambient temperature (a).

[0012] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness at 5.25 degrees C Desiccation ambient temperature (a) of a period with more solvent contents in a paint film than one half of the solvent contents in the paint film immediately after spreading is made below into the boiling point of the solvent contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this boiling point said more than desiccation ambient temperature (a).

[0013] Desiccation ambient temperature (a) of more periods than one half of the solvent contents in the paint film immediately after spreading of said desiccation ambient temperature of the solvent content in a paint film is made below into the glass transition point (Tg) of the binder contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film five publications characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this glass transition point (Tg) said more than desiccation ambient temperature (a).

[0014] The desiccation approach of the paint film characterized by heating-value  $Q \text{ kcal/m}^2$  which a shear rate gives to a paint film from a desiccation wind in 0.1sec-1 at the time of constant rate drying at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s at 50 micrometers or more of wet thickness, and hr being 50 - 1000 kcal/m<sup>2</sup> and hr at 6.25 degrees C.

[0015]  $Q(=h\Delta T')$ : The heating value h given to unit time amount and an unit area: Heat transfer coefficient (5-30kcal/m<sup>2</sup>, hr, and\*\*)

$\Delta T'$  (degree C): The desiccation approach of the paint film characterized by heating-value  $Q \text{ kcal/m}^2$  which a shear rate gives to a paint film from a desiccation wind in 0.1sec-1 at the time of constant rate drying at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s at 50 micrometers or more of wet thickness, and hr being 50 - 1000 kcal/m<sup>2</sup> and hr at 7.25 degrees C of differences of ambient temperature and paint film temperature.

[0016]  $Q(=h\Delta T')$ : The heating value h given to unit time amount and an unit area: Heat transfer coefficient (kcal/m<sup>2</sup>, hr, and\*\*)

deltaT' (degree C): The difference of ambient temperature and paint film temperature (10-50 degrees C) The desiccation approach of the paint film given in seven characterized by the heat transfer coefficients h of said heating value Q being 5-30kcal/m<sup>2</sup>, hr, and\*\*.

[0017] Hereafter, this invention is explained to a detail. The ease of flowing of a paint film is mentioned as a factor the paint film immediately after spreading turbulence-comes to be easy of a factor, this has the low viscosity of a paint film, and turbulence becomes strong when the thickness of a paint film is thick. Moreover, it is easy to produce turbulence the early stages of desiccation with many amounts of solvents in a paint film.

[0018] This invention is aimed at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa·s - 100 Pa·s by 50 micrometers or more of wet thickness by 25 degrees C which is comparatively easy to produce turbulence of a paint film, and the shear rate in 0.1sec-1.

[0019] When the 1st invention makes (D) g/m<sup>2</sup> the solvent content in the paint film in a certain time of day under g/m (D0)<sup>2</sup> and desiccation for the solvent content in the paint film immediately after spreading in the desiccation process which dries the above-mentioned paint film, It is characterized by the reduction rate (deltaD/D0) / deltat of solvent content until 95 mass % of D0 dries from a paint film being 0.01-0.06sec-1.

[0020] deltat is the difference of a certain time of day t1 and t2 (t1<t2) under desiccation, and t2-t1 here, and deltaD is the solvent content variation in the paint film between delta t.

[0021] When the reduction rate of solvent content is larger than 0.06sec-1, a rate of drying is too quick, and the mottle nonuniformity by turbulence of the paint film accompanying the solvent migration in a paint film arises. Conversely, when a reduction rate is smaller than 0.01sec-1, the drying time is needed greatly, a desiccation process becomes long too much, and the addition like a facility becomes large. As desirable range of the reduction rate (deltaD/D0) / deltat of solvent content, it is 0.01-0.04sec-1.

[0022] In invention of \*\*\*\* 1, it is desirable that absolute value |delta T/delta t| of the change rate of paint film temperature until 95 mass % of the solvent in said paint film dries from a paint film is 0.5-7 degrees C/sec. In addition, deltaT is the amount of paint film temperature changes between delta t.

[0023] When the absolute value of a change rate was less than 0.5 degrees C/sec, where turbulence of a paint film is suppressed, if productive efficiency exceeds 7 degrees C [ past / the low one / and ]/sec, control of turbulence of a paint film will become impossible easily.

[0024] 2nd invention is characterized by setting up ambient temperature until 95 mass % of the solvent in a paint film dries from a paint film among 20-85 degrees C, establishing at least 20-degree C temperature gradient, and making it go up gradually in the desiccation process which dries the above-mentioned paint film.

[0025] Since the mottle nonuniformity by turbulence of a paint film will arise if a rate of drying is high in the situation that a paint film tends to flow, as for the solvent evaporation in early stages of desiccation, it is desirable [ there are many amounts of solvents in a paint film, and ] to make drying temperature low so that it may not become not much high, and if the amount of solvents in a paint film decreases and floating of a paint film decreases, in order to raise drying efficiency, it is desirable to make drying temperature high. Although desirable temperature changes with the amounts of solvents and solvent kinds in the paint film immediately after spreading in drying temperature, while ambient temperature is 20-85 degrees C, it is desirable that it is, and, as for a temperature gradient until 95 mass % of the solvent in the early stages of desiccation and a paint film dries from a paint film, it is desirable to prepare 20 degrees C or more. "Ambient temperature" here points out the temperature of the space by which temperature control is carried out, in order to control desiccation. The failure called brushing produced when paint film temperature falls from a surrounding dew-point with the latent heat which a rate of drying will become remarkably slow if ambient temperature is lower than 20 degrees, and is taken by evaporation of the solvent not only from productivity worsening but a paint film and dew condensation takes place on a paint film may arise. On the other hand, if ambient temperature is higher than 85 degrees C, the vapor rate of the solvent from a paint film will be too quick, and turbulence of a paint film will arise. Moreover, when a temperature gradient is less than 20 degrees C, it is difficult to

reconcile control and productive efficiency of turbulence. [ paint film ]

[0026] In raising drying temperature gradually, 3rd invention is characterized by absolute value  $|\Delta T/\Delta t|$  of the change rate of paint film temperature until 95 mass % of the solvent in a paint film dries from a paint film being 0.5-7 degrees C/sec in the desiccation process which dries the above-mentioned paint film. When rate of change was less than 0.5 degrees C/sec, where turbulence of a paint film is suppressed, if productive efficiency exceeds 7 degrees C [ past / the low one / and ]/sec, control of turbulence of a paint film cannot be performed. In invention of \*\*\*\* 3, it is desirable to set up ambient temperature until 95 mass % of the solvent in a paint film dries from a paint film further among 20-85 degrees C, to establish at least 20-degree C temperature gradient, and to make it go up gradually.

[0027] The 4th invention makes desiccation ambient temperature (a) of more periods than one half of the solvent contents in the paint film immediately after spreading of the solvent content in a paint film below the glass transition point  $T_g$  of the binder contained among a paint film more than the dew-point temperature in the desiccation process which dries the above-mentioned paint film. [ most ] The solvent content in a paint film is characterized by making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below into the above-mentioned glass transition point  $T_g$  said more than desiccation ambient temperature (a).

[0028] In this invention, it is required to make desiccation ambient temperature (a) and (b) lower than  $T_g$  of the binder contained among a paint film. [ most ] When desiccation ambient temperature is high and paint film temperature exceeds  $T_g$  of the above-mentioned binder, it softens and it becomes impossible for a binder to form a uniform film surface. Moreover, it is necessary to make paint film temperature higher as mentioned above than the dew-point of a circumference ambient atmosphere.

[0029] Therefore, when raising desiccation ambient temperature gradually, desiccation ambient temperature (a) in a period with more solvent contents in a paint film than one half of the solvent content whole quantity in the paint film immediately after spreading is made below into the glass transition point  $T_g$  contained among a paint film more than the dew-point temperature. [ most ] By setting up the desiccation ambient temperature (b) in the event of the solvent content in a paint film becoming 1/2 or less [ of the solvent content whole quantity in the paint film immediately after spreading ] so that it may become below the above-mentioned glass transition point  $T_g$  more than desiccation ambient temperature (a), a paint film good [ without making a paint film produce turbulence ] can be formed.

[0030] The 5th invention makes desiccation ambient temperature (a) of more periods than one half of the solvent contents in the paint film immediately after spreading of the solvent content in a paint film below the boiling point  $T_b$  of the solvent contained among a paint film more than the dew-point temperature in the desiccation process which dries the above-mentioned paint film. [ most ] The solvent content in a paint film is the desiccation approach which makes 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below the boiling point  $T_b$  of the solvent contained among a paint film more than desiccation ambient temperature (a). [ most ]

[0031] Desiccation ambient temperature needs to carry out to below the boiling point of the solvent contained among a paint film. [ most ] If desiccation ambient temperature is high and paint film temperature exceeds the boiling point of a solvent, ebullition will take place within a paint film and generating of air bubbles and destruction of a paint film will occur. Therefore, desiccation ambient temperature (a) when the amount of solvents remains to 1/2 is set up so that it may become below  $T_b$  more than the dew-point temperature, and when the amount of solvents becomes 1/2 or less, it can obtain a paint film good [ without producing turbulence of a paint film ] by carrying out temperature setting out of desiccation conditions so that it may become below  $T_b$  more than desiccation ambient temperature (a).

[0032] As for the factor which produces turbulence of the paint film at the time of constant rate drying, the clearance rate of the solvent from a motion and paint film of the air near the paint film is mentioned. These relation is given by the degree type as a formula of the heating value given to a paint film for desiccation.

[0033] Heating-value  $Q = h\Delta T'Q$  (kcal/m<sup>2</sup>, hr): \*\*\*\* of the heating-value  $h$ (kcal/m<sup>2</sup>, hr, and \*\*):heat transfer coefficient  $\Delta T'$ (degree-C):ambient temperature given to unit time amount and an unit area

and paint film temperature and  $h$  express a film coefficient of heat-transfer, when telling heat from a gaseous phase to a paint film.

[0034] The above-mentioned formula changes a value by the flow of the air near the paint film, and can be called factor about turbulence of the paint film by motion of air. Substantially,  $\Delta T'$  (degree C) is a value decided by drying temperature, and can be called factor about the clearance rate of a solvent.

[0035] In the desiccation process at which invention of the 6th and 7 dries the above-mentioned paint film A paint film front face a fluidity It is the desiccation approach which set to 50 - 1000 kcal/m<sup>2</sup> and hr the heating value Q given to a paint film from a desiccation wind at the time of the constant rate drying to which desiccation advances in the condition of having had. A heat transfer coefficient  $h$  difference  $\Delta T$  [ of 5-30kcal/m<sup>2</sup>, hr and\*\* or ambient temperature, and paint film temperature ] '(degree C) It is characterized by what was specified at 10-50 degrees C.

[0036] In order not to produce turbulence of a paint film and to acquire sufficient productivity, it is attained by setting to 50 - 1000 kcal/m<sup>2</sup> and hr the heating value Q given to a paint film from a desiccation wind at the time of constant rate drying, and they are 100 - 900 kcal/m<sup>2</sup> and hr more preferably.

[0037] Moreover, as for the value of  $h$ , it is desirable to consider as 5-30kcal/m<sup>2</sup>, hr, and\*\*, and it is more desirable to consider as 5-20kcal/m<sup>2</sup>, hr, and\*\*. Furthermore, as for the value of  $\Delta T'$ , it is desirable to consider as 10-50 degrees C, and it is more desirable to consider as 10-40 degrees C.

[0038] If what distributed the dissolution or a solid is applied on a base material (coating) and this invention may dry a solute in a solvent, a solvent kind, a solute kind, and a solid kind are not limited, and when carrying out dissolution coating of the giant-molecule binder to what carries out coating of the gelatin water solution represented by photosensitive material, a solvent uses an organic solvent in many cases. Although some which take such a gestalt have many coating of a magnetic material, coating of the sensitization layer of a PS plate, etc., the feeling agent of heat developing is mentioned as a thing in viscosity range like this invention, and the spreading thickness range.

[0039] This invention is explained below and it explains based on drawing. Drawing 1 is the outline sectional view having shown signs that the paint film formed on the base material was dried, being conveyed in a desiccation box.

[0040] Generally desiccation follows the following progress from coating. The base material 3 \*\*\*\*\*(ed) from the former volume is sent to the die coating machine 5, and a paint film is applied to it. As for a base material 3, it is desirable to clean a spreading side, a rear face, or both sides with a web cleaner just before coating. If coating liquid is applied while the foreign matter had adhered to the spreading side, the function for which a foreign matter enters into a paint film and it asks will no longer be obtained, and also it may become the cause of the muscle failure in spreading. If it applies while the foreign matter had adhered to the rear face with the spreading gestalt which has a back roll, a base material will be lifted with a foreign matter at the time of spreading, and it becomes impossible to obtain the spreading thickness for which it asks locally. An adhesion roll, spraying of clean air, etc. are used as a web cleaner.

[0041] Since when it is not limited in this invention, roll coaters, such as a double roll coater and a reverse roll coater, a gravure coating machine, a comma coating machine, an extrusion die coating machine, etc. mention and \*\*\*\* forms the paint film of a thick film comparatively like this invention prevents that the paint film after coating flows a base material top with gravity, a spreading means is desirable for the conveyance direction of the base material just behind coating being more close to a horizontal from gravity. Although a paint film may be suitable in the gravity direction more than perpendicularity [ the paint film just behind coating ], or it with both physical relationship when a back roll exists using a die coating machine etc., the horizontally near thing of the base material conveyance angle to degree process is desirable after that.

[0042] Although there are some which used infrared radiation and microwave in desiccation, many means to evaporate a solvent from a paint film are used by generally spraying at homogeneity heating and the air which carried out gas conditioning on a paint film. Moreover, in order to repair the solvent which evaporated efficiently and to control a desiccation environment, a desiccation process is made in

the shape of a box. At this time, although \*\*\*\*\* [ the number of the desiccation boxes 1 / one ], the desiccation conditions of each zone can be selectively changed by dividing into two or more desiccation zones 2. When spraying air on a paint film and drying it, as desiccation conditions for each zone, drying temperature, the rate of the air to spray, the diffuser of the air to spray and the distance of a paint film, the amount of supply of a fresh air content, etc. are mentioned. In order to spray supply of air in a desiccation zone on a paint film, another feed hopper which cannot do effect of the flow of air easily to the paint film other than the air from a nozzle 4 (or slit) etc. may be used together. Since it will be easy to disturb a paint film if there are many amounts of the air sprayed on a paint film on the conditions which the mottle nonuniformity made into the problem especially by this invention tends to generate, it is important to prepare another feed hopper for supply of sufficient fresh air into the desiccation box 1. When using an organic solvent as a solvent of a paint film, generally supervising the solvent steamy concentration in the desiccation box 1 is performed. When the desiccation box 1 is divided into two or more desiccation zones 2, it supervises for every zone. When conveying supporting a base material 3 with a backup roll from a rear face within a dryer, as for the degree of \*\*\* of a backup roll, it is common to be referred to as 100 micrometers or less, and it is desirable to be referred to as 50 micrometers or less. A paint film has not been dried from the die coating machine 5 up to the inside of the desiccation box 1, and since the foreign matter adhering to a front face is pasted up with a paint film, it is necessary to keep a surrounding air cleanliness class high. Although the air cleanliness class in the desiccation box 1 is based also on the character of the product to manufacture, it is desirable to consider as 100 or less class preferably 1000 or less class. Moreover, it is necessary to isolate the fan and the source of a shock for especially keeping away the source of raising dust and supplying air in the desiccation box 1 from the desiccation box 1 around desiccation box 1.

[0043] To affect the function of a product in which the amount of residual solvents in a paint film was finished, it is necessary to control, and the heat cure zone for applying heat is usually prepared after passing through the desiccation box 1. It may be continuously connected in the condition of having heated, between the desiccation box 1 and the heat cure zone, and it may be cooled ordinary temperature or if needed once. Two or more rolls can also perform conveyance of the base material in a heat cure zone, and by the air float method, a base material may be surfaced and may be conveyed. Since a heat cure zone has a usually long conveyance system way, it is desirable to form the web guide system for controlling the drive roll and base material conveyance location for base material conveyance in order.

[0044]

[Example] Although an example is given and this invention is hereafter explained to a detail, the mode of this invention is not limited to this.

[0045] The giant-molecule binder was dissolved in the production methyl ethyl ketone (MEK) of a spreading sample by solid content concentration 25 mass %, and the shear rate obtained the coating liquid of viscosity 150 mPa·s in 0.1s-1 at 25 degrees C. Coating of this coating liquid was carried out to homogeneity by spreading rate 50 m/min so that it might become 100 micrometers of wet thickness on a 100-micrometer PET base material by the die coating machine.

[0046] This base material by which coating was carried out from the die coating machine was conveyed to the desiccation box which consists of three desiccation zones like drawing 1, it dried by spraying from a slit heating and the air by which gas conditioning was carried out on a paint film front face, and the spreading sample was obtained. The following assessment was performed about the obtained spreading sample.

[0047] (Assessment)

- A mottle nonuniformity generating situation is what carried out observation assessment by viewing, and 10 is [ 1 is bad and ] good at ten-step assessment.

[0048] About the spreading sample which is the example 1 above, and was made and obtained, desiccation ambient temperature of three desiccation zones of a desiccation box was made into 40 degrees C also with each zone, the strength of the desiccation style was changed, and samples 1 and 2 were obtained. The sample after spreading and predetermined time carries out the quick stop of the conveyance of a base material, and is sampled, and the value which analyzed MEK which remains in a

sample about the solvent content D in a paint film (the amount of residual solvents) with the gas chromatography is shown in a table 1 (sample 1) and a table 2 (sample 2). Moreover,  $(\Delta D/D_0)/\Delta t$  was shown similarly. In addition, the solvent content D0 immediately after spreading (first stage) was 85 g/m<sup>2</sup>.

[0049]

[A table 1]

塗布後の時間(sec)	5	10	15	20	25	30	35	40
D(g/m <sup>2</sup> )	78.6	70.2	58.6	46.5	33.9	22.1	10.6	0.01 以下
$(1 - (D/D_0)) \times 100(\%)$	8%	17%	31%	45%	60%	74%	88%	—
$(\Delta D/D_0)/\Delta t(\text{sec}^{-1})$	0.0151	0.0198	0.0273	0.0285	0.0296	0.0278	0.0271	—

[0050]

[A table 2]

塗布後の時間(sec)	5	10	15	20
D(g/m <sup>2</sup> )	74.8	44.3	14.2	0.01 以下
$(1 - (D/D_0)) \times 100(\%)$	12%	48%	83%	—
$(\Delta D/D_0)/\Delta t(\text{sec}^{-1})$	0.0240	0.0718	0.0708	—

[0051] Since it had not dried at a dryer outlet when set to  $(\Delta D/D_0)/\Delta t < 0.01 \text{ sec}^{-1}$ , it was not suitable for practical use to become an experiment termination etc. On the other hand about mottle nonuniformity, the number of mottle nonuniformity of the sample 1 was three by ten points and the sample 2.

[0052] It was made to dry on each desiccation conditions of a table 3 which show below the spreading sample which is the example 2 above, and was made and obtained, and the mottle nonuniformity of the obtained sample was evaluated.

[0053]

[A table 3]

試料	第1ゾーン	第2ゾーン	第3ゾーン	モトルムラレベル
3	40°C	60°C	80°C	10
4	20°C	30°C	40°C	ブラッシング、未乾
5	40°C	50°C	60°C	未乾
6	60°C	70°C	80°C	6
7	40°C	70°C	90°C	8

[0054] Since the 20-degree C temperature gradient was established among 20-85 degrees C and drying temperature was raised gradually, generating of mottle nonuniformity is suppressed, and it turns out that a sample 3 is good so that clearly from a table 3. On the other hand, like samples 4 and 5, at low temperature, the temperature rise put 10 degrees C, the 1st zone suited, the drying time started too much, and desiccation was not completed, but the case became wetness. And as for the sample 4, generating of brushing was also accepted. Moreover, the 1st zone is made into 60 degrees C like a sample 6, when a temperature rise is 10-degree-C difference, mottle nonuniformity does not improve, and also when ambient temperature exceeds 85 degrees C like a sample 7, mottle nonuniformity cannot fully be said to be fitness.

[0055] About the spreading sample which is the example 3 above, and was made and obtained, desiccation ambient temperature of three desiccation zones of a desiccation box was made into 40 degrees C also with each zone, the strength of the desiccation style was changed, samples 8 and 9 were obtained, and the temperature of the paint film was measured with the non-contact-type radiation

thermometer. Measurement was measured in the location through which it passes after the predetermined time after spreading, and the location through which it passes after [ of a there ] 0.5 seconds, and computed  $|\Delta T/\Delta t|$  from the temperature gradient of two points, and time difference (0.5 seconds). The value which analyzed MEK which remains in a sample with the gas chromatography about the solvent content D in a paint film (the amount of residual solvents) was shown in a table 4 (sample 8) and a table 5 (sample 9). Moreover,  $(\Delta D/D_0)/\Delta t$  was shown similarly. In addition, the early solvent content  $D_0$  was 85 g/m<sup>2</sup>.

[0056]

[A table 4]

塗布後の時間(sec)	5	10	15	20	25	30	35	40
D(g/m <sup>2</sup> )	78.6	70.2	58.6	46.5	33.9	22.1	10.6	0.01 以下
(1 - (D/D <sub>0</sub> )) × 100(%)	8%	17%	31%	45%	60%	74%	88%	-
ΔT/Δt(°C/sec)	6.1	2.9	1.8	1.2	0.9	0.7	0.6	-

[0057]

[A table 5]

塗布後の時間(sec)	5	10	15	20
D(g/m <sup>2</sup> )	74.8	44.3	14.2	0.01 以下
(1 - (D/D <sub>0</sub> )) × 100(%)	12%	48%	83%	-
ΔT/Δt(°C/sec)	8.6	4.9	1.4	-

[0058] The sample 8 which has  $|\Delta T/\Delta t|$  in the range of 0.5-7 degrees C/sec was excellent, and, on the other hand, the number of ten mottle nonuniformity of a sample 9 of mottle nonuniformity was three so that clearly from a table 4 and a table 5.

[0059] It changed into the giant-molecule binder with which example 4 glass transition points differ, and also samples 10-13 were produced in the same procedure as production of a spreading sample. The dew-point temperature at this time was 13 degrees C. It was made to dry on each desiccation conditions of a table 6 which show this below, and the mottle nonuniformity of the obtained sample was evaluated.

[0060]

[A table 6]

試料	バインダー Tg(°C)	溶媒量1/2までの 乾燥雰囲気温度(°C)	溶媒量1/2以下の 乾燥雰囲気温度(°C)	モトルムラ レベル
10	72	30	60	10
11	72	10	60	ブラッシング
12	72	30	75	3
13	85	30	80	4

[0061] In making [ the desiccation approach ] with below the glass transition point Tg of a binder, and an amount [ of solvents ] of 1/2 or less desiccation ambient temperature below into Tg of a binder for the desiccation ambient temperature to the amounts 1/2 of solvents more than previous desiccation ambient temperature more than the dew-point temperature like a sample 10, generating of mottle nonuniformity was suppressed and was a good result so that clearly from a table 6. In the case where with an amount [ of solvents ] of 1/2 or less desiccation ambient temperature exceeds the glass transition point of a binder for brushing like a lifting and a sample 12 in the case where desiccation ambient temperature to the amounts 1/2 of solvents is made below into the dew-point temperature like a sample 11 on the other hand, mottle nonuniformity got worse greatly. Furthermore, when with an amount [ of solvents ] of 1/2 or less desiccation ambient temperature exceeded the boiling point of MEK (79.6 degrees C) like the sample 13, mottle nonuniformity got worse remarkably and a cellular hole from which gas escaped to the paint film generated it.

[0062] It was made to dry on each desiccation conditions of a table 7 which show below the spreading sample which is the example 5 above, and was made and obtained, and the mottle nonuniformity of the obtained samples 14-18 was evaluated.  $\Delta T'$  considers as the difference of the paint film temperature measured with the non-contact-type radiation thermometer, and whenever [ desiccation box internal temperature ], and since paint film temperature changes with desiccation, it has width of face. The following conditions are conditions until constant rate drying is completed, and the terminal point of constant rate drying was checked because it touches and is dry on the paint film front face. The drying time in a table was expressed with what time of time amount which the sample 14 took the time amount which each sample took even at the desiccation-after spreading - terminal point it becomes.

[0063]

[A table 7]

試料	Q (kcal/m <sup>2</sup> ·hr)	h (kcal/m <sup>2</sup> ·hr·°C)	$\Delta T'$ (°C)	モトルムラ レベル	乾燥時間
14	340～440	20	17～22	10	1
15	680～940	40	17～24	9	0.6
16	60～240	30	2～9	10	1.7
17	34～40	2	17～20	10	9
18	1050～1230	30	35～41	3	0.4

[0064] By the sample 14, all of difference  $\Delta T'$  of a heating value Q, a heat transfer coefficient h, and ambient temperature and paint film temperature were within the limits of this invention enclosure, and mottle nonuniformity was good so that clearly from a table 7. By the sample 15, since h is high, some lowering is looked at by mottle nonuniformity, and by the sample 16, since  $\Delta T'$  is low, the drying time is long a little. By the sample 17, since Q and h were low, although mottle nonuniformity was good, the drying time was too long, productivity got worse remarkably, and by the sample 18, since Q was too large, mottle nonuniformity got worse remarkably.

[0065]

[Effect of the Invention] The notably excellent effectiveness that the paint film front face which controls mottle nonuniformity and has the outstanding smooth nature can be formed is done so by controlling the rate of drying of the paint film from which the shear rate applied and obtained coating liquid at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa·s - 100 Pa·s by 50 micrometers or more of wet thickness in 0.1sec-1 by 25 degrees C according to this invention.

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[Translation done.]

**\* NOTICES \***

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**TECHNICAL FIELD**

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[Field of the Invention] This invention relates to the technique which dries while controlling especially a rate of drying about the desiccation approach of a paint film of having applied and obtained coating liquid.

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[Translation done.]

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**CLAIMS**

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**[Claim(s)]**

[Claim 1] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness at 25 degrees C When the solvent content in the paint film in a certain time of day under g/m (D0)2 and desiccation is made into (D) g/m<sup>2</sup> for the solvent content in the paint film immediately after spreading, The desiccation approach of the paint film characterized by the reduction rate ( $\Delta D/D_0$ ) /  $\Delta t$  of solvent content until 95 mass % of D0 dries from a paint film being 0.01-0.06sec-1.

$\Delta t$ : Solvent content variation in the difference of a certain time of day t1 and t2 ( $t_1 < t_2$ ) under desiccation, and the paint film between  $t_2 - t_1$   $\Delta D/\Delta t$  [claim 2] The desiccation approach of the paint film according to claim 1 characterized by absolute value  $|\Delta T/\Delta t|$  of the change rate of paint film temperature until 95 mass % of the solvent in said paint film dries from a paint film being 0.5-7 degrees C/sec.

The amount of paint film temperature changes between  $\Delta T/\Delta t$  [claim 3] The desiccation approach of the paint film characterized by being while ambient temperature until 95 mass % of the solvent in a paint film dries from a paint film is 20-85 degrees C, and establishing at least 20-degree C temperature gradient, and making it go up gradually at the desiccation process at which a shear rate dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s at 50 micrometers or more of wet thickness in 0.1sec-1 at 25 degrees C.

[Claim 4] The desiccation approach of the paint film characterized by absolute value  $|\Delta T/\Delta t|$  of the change rate of paint film temperature until 95 mass % of the solvent in a paint film dries from a paint film being 0.5-7 degrees C/sec at the desiccation process at which a shear rate dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s at 50 micrometers or more of wet thickness in 0.1sec-1 at 25 degrees C.

[Claim 5] The desiccation approach of the paint film according to claim 4 which is while ambient temperature until 95 mass % of the solvent in said paint film dries from a paint film is 20-85 degrees C, and is characterized by establishing at least 20-degree C temperature gradient, and making it go up gradually.

[Claim 6] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness at 25 degrees C Desiccation ambient temperature (a) of a period with more solvent contents in a paint film than one half of the solvent contents in the paint film immediately after spreading is made below into the glass transition point (Tg) of the binder contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this glass transition point (Tg) said more than desiccation ambient temperature (a).

[Claim 7] A shear rate at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa-s - 100 Pa-s in 0.1sec-1 at 50 micrometers or more of wet thickness at 25

degrees C Desiccation ambient temperature (a) of a period with more solvent contents in a paint film than one half of the solvent contents in the paint film immediately after spreading is made below into the boiling point of the solvent contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this boiling point said more than desiccation ambient temperature (a).

[Claim 8] Desiccation ambient temperature (a) of more periods than one half of the solvent contents in the paint film immediately after spreading of said desiccation ambient temperature of the solvent content in a paint film is made below into the glass transition point (Tg) of the binder contained among a paint film more than the dew-point temperature. [ most ] The desiccation approach of the paint film according to claim 7 characterized by the solvent content in a paint film making 1/2 or less [ of the solvent content in the paint film immediately after spreading ] desiccation ambient temperature (b) below this glass transition point (Tg) said more than desiccation ambient temperature (a).

[Claim 9] The desiccation approach of the paint film characterized by heating-value Qkcal/m<sup>2</sup> which a shear rate gives to a paint film from a desiccation wind in 0.1sec<sup>-1</sup> at the time of constant rate drying at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa·s - 100 Pa·s at 50 micrometers or more of wet thickness, and hr being 50 - 1000 kcal/m<sup>2</sup> and hr at 25 degrees C.

Q(=h $\Delta$ T'): The heating value h given to unit time amount and an unit area: Heat transfer coefficient (5-30kcal/m<sup>2</sup>, hr, and\*\*)

$\Delta$ T' (degree C): The difference of ambient temperature and paint film temperature [claim 10] The desiccation approach of the paint film characterized by heating-value Qkcal/m<sup>2</sup> which a shear rate gives to a paint film from a desiccation wind in 0.1sec<sup>-1</sup> at the time of constant rate drying at the desiccation process which dries the paint film to which viscosity applied the coating liquid of 0.5 mPa·s - 100 Pa·s at 50 micrometers or more of wet thickness, and hr being 50 - 1000 kcal/m<sup>2</sup> and hr at 25 degrees C.

Q(=h $\Delta$ T'): The heating value h given to unit time amount and an unit area: Heat transfer coefficient (kcal/m<sup>2</sup>, hr, and\*\*)

$\Delta$ T' (degree C): The difference of ambient temperature and paint film temperature (10-50 degrees C)

[Claim 11] The desiccation approach of the paint film according to claim 10 characterized by the heat transfer coefficients h of said heating value Q being 5-30kcal/m<sup>2</sup>, hr, and\*\*.

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(54) 【発明の名称】 塗膜の乾燥方法

(57) 【要約】

【課題】 25°Cで剪断速度が0.1 sec<sup>-1</sup>において粘度が0.5 mPa·s~100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程にて、塗布液を塗布して得た塗膜の乾燥速度を制御することによりモトルムラを抑制し、優れた平滑性を有する塗膜表面を形成すること。

【解決手段】 25°Cで剪断速度が0.1 sec<sup>-1</sup>において粘度が0.5 mPa·s~100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗布直後の塗膜中の溶媒含有量を(D<sub>0</sub>) g/m<sup>2</sup>、乾燥中のある時刻での塗膜中の溶媒含有量を(D) g/m<sup>2</sup>としたとき、D<sub>0</sub>の95質量%が塗膜から乾燥するまでの間の溶媒含有率の減少速度(△D/D<sub>0</sub>)/△tが0.01~0.06 sec<sup>-1</sup>であることを特徴とする塗膜の乾燥方法。

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## 【特許請求の範囲】

【請求項1】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗布直後の塗膜中の溶媒含有量を(D<sub>0</sub>) g/m<sup>2</sup>、乾燥中のある時刻での塗膜中の溶媒含有量を(D) g/m<sup>2</sup>としたとき、D<sub>0</sub>の95質量%が塗膜から乾燥するまでの間の溶媒含有率の減少速度(△D/D<sub>0</sub>)/△tが0. 01～0. 06 sec<sup>-1</sup>であることを特徴とする塗膜の乾燥方法。

△t : 乾燥中のある時刻t<sub>1</sub>、t<sub>2</sub>(t<sub>1</sub><t<sub>2</sub>)の差、t<sub>2</sub>-t<sub>1</sub>

△D : △t間の塗膜中の溶媒含有量変化量

【請求項2】 前記塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値|△T/△t|が0. 5～7°C/secであることを特徴とする請求項1記載の塗膜の乾燥方法。

△T : △t間の塗膜温度変化量

【請求項3】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の雰囲気温度が20～85°Cの間であり、かつ少なくとも20°Cの温度差を設けて段階的に上昇させることを特徴とする塗膜の乾燥方法。

【請求項4】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値|△T/△t|が0. 5～7°C/secであることを特徴とする塗膜の乾燥方法。

【請求項5】 前記塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の雰囲気温度が20～85°Cの間であり、かつ少なくとも20°Cの温度差を設けて段階的に上昇させることを特徴とする請求項4記載の塗膜の乾燥方法。

【請求項6】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥雰囲気温度

(a) を露点温度以上塗膜中最多く含まれるバインダーのガラス転移点(T<sub>g</sub>)以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥雰囲気温度(b)を前記乾燥雰囲気温度(a)以上同ガラス転移点(T<sub>g</sub>)以下とすることを特徴とする塗膜の乾燥方法。

【請求項7】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液

をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥雰囲気温度

(a) を露点温度以上塗膜中最多く含まれる溶媒の沸点以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥雰囲気温度(b)を前記乾燥雰囲気温度(a)以上同沸点以下とすることを特徴とする塗膜の乾燥方法。

【請求項8】 前記乾燥雰囲気温度を、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥雰囲気温度(a)を露点温度以上塗膜中最多く含まれるバインダーのガラス転移点(T<sub>g</sub>)以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥雰囲気温度(b)を前記乾燥雰囲気温度(a)以上同ガラス転移点(T<sub>g</sub>)以下とすることを特徴とする請求項7記載の塗膜の乾燥方法。

【請求項9】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、恒率乾燥時に乾燥風から塗膜に与える熱量Q kcal/m<sup>2</sup>·hrが50～1000 kcal/m<sup>2</sup>·hrであることを特徴とする塗膜の乾燥方法。

Q (= h ΔT') : 単位時間、単位面積に与える熱量  
h : 伝熱係数(5～30 kcal/m<sup>2</sup>·hr·°C)

ΔT' (°C) : 雰囲気温度と塗膜温度の差

【請求項10】 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa·s～100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、恒率乾燥時に乾燥風から塗膜に与える熱量Q kcal/m<sup>2</sup>·hrが50～1000 kcal/m<sup>2</sup>·hrであることを特徴とする塗膜の乾燥方法。

Q (= h ΔT') : 単位時間、単位面積に与える熱量  
h : 伝熱係数(kcal/m<sup>2</sup>·hr·°C)

ΔT' (°C) : 雰囲気温度と塗膜温度の差(10～50°C)

【請求項11】 前記熱量Qの伝熱係数hが5～30 kcal/m<sup>2</sup>·hr·°Cであることを特徴とする請求項10記載の塗膜の乾燥方法。

【発明の詳細な説明】

40 【0001】

【発明の属する技術分野】本発明は塗布液を塗布して得た塗膜の乾燥方法に関し、特に乾燥速度を制御しながら乾燥を行う技術に関する。

【0002】

【従来の技術】塗布液を塗布して得た塗膜を乾燥する方法として、例えば特開昭50-20355号に、非セット系塗布組成物を塗布して得た写真感光材料の初期乾燥時の温度を30～100°Cの温度範囲より選択し、後期乾燥時の乾燥温度を50～150°Cの温度範囲であり、

50 かつ初期乾燥温度よりも20～50°C高く設定する方法

が開示されている。

【0003】

【発明が解決しようとする課題】支持体上に塗布された塗膜を乾燥させる手段として広く用いられる加熱した空気を吹き付ける手段では、吹き付けた空気により塗膜表面が乱され表面の平滑度を失ういわゆるモトルムラを生じる。従って塗膜に到達する空気の流速を抑えることによりこのモトルムラを弱くすることが出来るが、塗膜からの溶媒の除去速度が速いと塗膜内の溶媒移動による流動と、塗膜から出た溶媒の滞留により塗膜表面が乱され、先と同様にモトルムラを生じる。

【0004】本発明は上記事情に鑑みて為されたものであり、その目的は塗布液を塗布して得た塗膜の乾燥速度を制御することによりモトルムラを抑制し、優れた平滑性を有する塗膜表面を形成することにある。

【0005】

【課題を解決するための手段】本発明の上記目的は以下の構成により達成された。

【0006】1. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗布直後の塗膜中の溶媒含有量を

(D<sub>0</sub>) g/m<sup>2</sup>、乾燥中のある時刻での塗膜中の溶媒含有量を(D) g/m<sup>2</sup>としたとき、D<sub>0</sub>の95質量%が塗膜から乾燥するまでの間の溶媒含有率の減少速度(ΔD/D<sub>0</sub>) / Δt が0. 01 ~ 0. 06 sec<sup>-1</sup>であることを特徴とする塗膜の乾燥方法。

【0007】Δt : 乾燥中のある時刻 t<sub>1</sub>、t<sub>2</sub> (t<sub>1</sub> < t<sub>2</sub>) の差、t<sub>2</sub> - t<sub>1</sub>

ΔD : Δt 間の塗膜中の溶媒含有量変化量

前記塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値 |ΔT/Δt| が0. 5 ~ 7°C/sec であることを特徴とする1記載の塗膜の乾燥方法。

【0008】ΔT : Δt 間の塗膜温度変化量

2. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の霧囲気温度が20 ~ 85°Cの間であり、かつ少なくとも20°Cの温度差を設けて段階的に上昇させることを特徴とする塗膜の乾燥方法。

【0009】3. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値 |ΔT/Δt| が0. 5 ~ 7°C/sec であることを特徴とする塗膜の乾燥方法。

【0010】前記塗膜中の溶媒の95質量%が塗膜から

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乾燥するまでの間の霧囲気温度が20 ~ 85°Cの間であり、かつ少なくとも20°Cの温度差を設けて段階的に上昇させることを特徴とする3記載の塗膜の乾燥方法。

【0011】4. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥霧囲気温度(a)を露点温度以上塗膜中最多く含まれるバインダーのガラス転移点(Tg)以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥霧囲気温度(b)を前記乾燥霧囲気温度(a)以上同ガラス転移点(Tg)以下とすることを特徴とする塗膜の乾燥方法。

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【0012】5. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥霧囲気温度(a)を露点温度以上塗膜中最多く含まれる溶媒の沸点以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥霧囲気温度(b)を前記乾燥霧囲気温度(a)以上同沸点以下とすることを特徴とする塗膜の乾燥方法。

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【0013】前記乾燥霧囲気温度を、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥霧囲気温度(a)を露点温度以上塗膜中最多く含まれるバインダーのガラス転移点(Tg)以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下での乾燥霧囲気温度(b)を前記乾燥霧囲気温度(a)以上同ガラス転移点(Tg)以下とすることを特徴とする5記載の塗膜の乾燥方法。

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【0014】6. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、恒率乾燥時に乾燥風から塗膜に与える熱量Q kcal/m<sup>2</sup> · hr が50 ~ 1000 kcal/m<sup>2</sup> · hr であることを特徴とする塗膜の乾燥方法。

【0015】Q (= h ΔT') : 単位時間、単位面積に与える熱量

h : 伝熱係数 (5 ~ 30 kcal/m<sup>2</sup> · hr · °C)

ΔT' (°C) : 霧囲気温度と塗膜温度の差

7. 25°Cで剪断速度が0. 1 sec<sup>-1</sup>において粘度が0. 5 mPa · s ~ 100 Pa · s の塗布液をウェット膜厚50 μm以上で塗布した塗膜を乾燥する乾燥工程で、恒率乾燥時に乾燥風から塗膜に与える熱量Q kcal/m<sup>2</sup> · hr が50 ~ 1000 kcal/m<sup>2</sup> · hr であることを特徴とする塗膜の乾燥方法。

【0016】Q (= h ΔT') : 単位時間、単位面積に与える熱量

50 与える熱量

$h$  : 伝熱係数 ( $\text{Kcal}/\text{m}^2 \cdot \text{hr} \cdot ^\circ\text{C}$ )

$\Delta T'$  ( $^\circ\text{C}$ ) : 霧囲気温度と塗膜温度の差 (10~50  $^\circ\text{C}$ )

前記熱量Qの伝熱係数hが5~30  $\text{Kcal}/\text{m}^2 \cdot \text{hr} \cdot ^\circ\text{C}$ であることを特徴とする7記載の塗膜の乾燥方法。

【0017】以下、本発明を詳細に説明する。塗布直後の塗膜が乱れやすくなる因子としては塗膜の流動し易さが挙げられ、これは塗膜の粘度が低く、塗膜の厚さが厚い場合に乱れは強くなる。又、乱れは塗膜中の溶媒量が多い乾燥初期ほど生じやすい。

【0018】本発明は比較的塗膜の乱れを生じやすい25  $^\circ\text{C}$ 、剪断速度が0.1  $\text{sec}^{-1}$ において粘度が0.5  $\text{mPa} \cdot \text{s}$ ~100  $\text{Pa} \cdot \text{s}$ の塗布液をウェット膜厚50  $\mu\text{m}$ 以上で塗布した塗膜を乾燥する乾燥工程を対象とする。

【0019】第1の発明は、上記塗膜を乾燥する乾燥工程において、塗布直後の塗膜中の溶媒含有量を( $D_0$ )  $\text{g}/\text{m}^2$ 、乾燥中のある時刻での塗膜中の溶媒含有量を( $D$ )  $\text{g}/\text{m}^2$ としたとき、 $D_0$ の95質量%が塗膜から乾燥するまでの間の溶媒含有率の減少速度( $\Delta D/D_0$ )/ $\Delta t$ が0.01~0.06  $\text{sec}^{-1}$ であることを特徴とするものである。

【0020】ここで $\Delta t$ は乾燥中のある時刻 $t_1$ 、 $t_2$ ( $t_1 < t_2$ )の差、 $t_2 - t_1$ であり、 $\Delta D$ は $\Delta t$ 間の塗膜中の溶媒含有量変化量である。

【0021】溶媒含有率の減少速度が0.06  $\text{sec}^{-1}$ より大きいと乾燥速度が速すぎ、塗膜内の溶媒移動に伴う塗膜の乱れによるモトルムラが生じる。逆に減少速度が0.01  $\text{sec}^{-1}$ より小さいと乾燥時間を大きく必要とし、乾燥工程が長くなり過ぎ設備的な付加が大きくなる。溶媒含有率の減少速度( $\Delta D/D_0$ )/ $\Delta t$ の好ましい範囲としては0.01~0.04  $\text{sec}^{-1}$ である。

【0022】又第1の発明においては、前記塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値 $|\Delta T/\Delta t|$ が0.5~7  $^\circ\text{C/sec}$ であることが好ましい。尚、 $\Delta T$ は $\Delta t$ 間の塗膜温度変化量である。

【0023】変化速度の絶対値が0.5  $^\circ\text{C/sec}$ を下回ると塗膜の乱れを抑えた状態では生産効率が低すぎ、又7  $^\circ\text{C/sec}$ を上回ると塗膜の乱れの抑制が出来にくくなる。

【0024】第2の発明は、上記塗膜を乾燥する乾燥工程において、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の霧囲気温度を20~85  $^\circ\text{C}$ の間に設定し、少なくとも20  $^\circ\text{C}$ の温度差を設けて段階的に上昇させることを特徴とするものである。

【0025】塗膜中の溶媒量が多く、塗膜が流動しやすい状況において乾燥速度が高いと塗膜の乱れによるモトルムラが生じるため乾燥初期の溶媒蒸発はあまり高くな

らないように乾燥温度を低くすることが好ましく、塗膜中溶媒量が少なくなり、塗膜の流動が少なくなれば乾燥効率を高めるために乾燥温度を高くすることが好ましい。乾燥温度は塗布直後の塗膜中の溶媒量や、溶媒種により好ましい温度は異なるが、霧囲気温度が20~85  $^\circ\text{C}$ の間であることが好ましく、乾燥初期と塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の温度差は20  $^\circ\text{C}$ 以上設けることが好ましい。ここでいう「霧囲気温度」とは、乾燥を制御するために温度制御されている空間の温度を指す。霧囲気温度が20度より低いと乾燥速度が著しく遅くなり生産性が悪くなるだけでなく、塗膜からの溶媒の蒸発により奪われる潜熱により塗膜温度が周辺の露点より低下し、塗膜上で結露が起こることにより生じるブレッシングと呼ばれる故障が生じることがある。一方霧囲気温度が85  $^\circ\text{C}$ より高いと、塗膜からの溶媒の蒸発速度が速すぎ塗膜の乱れが生じてしまう。又温度差が20  $^\circ\text{C}$ を下回ると塗膜の乱れの抑制と生産効率を両立させることは難しい。

【0026】第3の発明は、上記塗膜を乾燥する乾燥工程において、乾燥温度を徐々に上昇させるに当たり、塗膜中の溶媒の95質量%が塗膜から乾燥するまでの塗膜温度の変化速度の絶対値 $|\Delta T/\Delta t|$ が0.5~7  $^\circ\text{C/sec}$ であることを特徴とするものである。変化率が0.5  $^\circ\text{C/sec}$ を下回ると塗膜の乱れを抑えた状態では生産効率が低すぎ、又7  $^\circ\text{C/sec}$ を上回ると塗膜の乱れの抑制が出来ない。又第3の発明においては、更に塗膜中の溶媒の95質量%が塗膜から乾燥するまでの間の霧囲気温度を20~85  $^\circ\text{C}$ の間に設定し、少なくとも20  $^\circ\text{C}$ の温度差を設けて段階的に上昇させることが好ましい。

【0027】第4の発明は、上記塗膜を乾燥する乾燥工程において、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2より多い期間の乾燥霧囲気温度

(a) を露点温度以上塗膜中最も多く含まれるバインダーのガラス転移点Tg以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の1/2以下の乾燥霧囲気温度(b) を前記乾燥霧囲気温度(a) 以上上記ガラス転移点Tg以下とすることを特徴とするものである。

【0028】この発明においては、乾燥霧囲気温度(a)及び(b)を塗膜中最も多く含まれるバインダーのTgより低くすることが必要である。乾燥霧囲気温度が高く、塗膜温度が上記バインダーのTgを越えるとバインダーは軟化し、均一な膜面を形成することが出来なくなる。又、塗膜温度は前述のように周辺霧囲気の露点より高くする必要がある。

【0029】従って乾燥霧囲気温度を段階的に上昇させる場合、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量全量の1/2より多い期間での乾燥霧囲気温度(a)を露点温度以上塗膜中最も多く含まれるガラス転

移点  $T_g$  以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量全量の  $1/2$  以下となった時点での乾燥雰囲気温度 (b) を乾燥雰囲気温度 (a) 以上上記ガラス転移点  $T_g$  以下となるよう設定することにより、塗膜に乱れを生じさせずに良好な塗膜を形成することが出来る。

【0030】第5の発明は、上記塗膜を乾燥する乾燥工程において、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の  $1/2$  より多い期間の乾燥雰囲気温度

(a) を露点温度以上塗膜中最も多く含まれる溶媒の沸点  $T_b$  以下とし、塗膜中の溶媒含有量が塗布直後の塗膜中の溶媒含有量の  $1/2$  以下の乾燥雰囲気温度 (b) を乾燥雰囲気温度 (a) 以上塗膜中最も多く含まれる溶媒の沸点  $T_b$  以下とする乾燥方法である。

【0031】乾燥雰囲気温度は塗膜中最も多く含有される溶媒の沸点以下とする必要がある。乾燥雰囲気温度が高く、塗膜温度が溶媒の沸点を超えると塗膜内で沸騰が起こり、気泡の発生や塗膜の破壊が発生する。従って、溶媒量が  $1/2$  まで残留している場合の乾燥雰囲気温度

(a) は、露点温度以上  $T_b$  以下となるように設定し、溶媒量が  $1/2$  以下となった時点では乾燥雰囲気温度

(a) 以上  $T_b$  以下となるよう乾燥条件の温度設定をすることにより塗膜の乱れを生じさせずに良好な塗膜を得ることが出来る。

【0032】恒率乾燥時の塗膜の乱れを生じさせる因子は、塗膜近傍での空気の動きと塗膜からの溶媒の除去速度が挙げられる。これらの関係は乾燥のために塗膜へ与える熱量の式として次式で与えられる。

【0033】熱量  $Q = h \Delta T'$

$Q$  ( $\text{kcal}/\text{m}^2 \cdot \text{hr}$ ) : 単位時間、単位面積に与える熱量

$h$  ( $\text{kcal}/\text{m}^2 \cdot \text{hr} \cdot {}^\circ\text{C}$ ) : 伝熱係数

$\Delta T'$  ( ${}^\circ\text{C}$ ) : 雰囲気温度と塗膜温度の差

尚、 $h$  は気相から塗膜へ熱を伝える場合は境界伝熱係数を表す。

【0034】上記式は塗膜近傍の空気の流れにより値が変わるものであり、空気の動きによる塗膜の乱れに関する因子といえる。 $\Delta T'$  ( ${}^\circ\text{C}$ ) は実質的には乾燥温度により決まる値であり、溶媒の除去速度に関する因子といえる。

【0035】第6及び7の発明は、上記塗膜を乾燥する乾燥工程において、塗膜表面が流動性を持った状態で乾燥が進行する恒率乾燥時に乾燥風から塗膜に与える熱量  $Q$  を  $50 \sim 1000 \text{ kcal}/\text{m}^2 \cdot \text{hr}$  とした乾燥方法であって伝熱係数  $h$  を  $5 \sim 30 \text{ kcal}/\text{m}^2 \cdot \text{hr} \cdot {}^\circ\text{C}$  或いは雰囲気温度と塗膜温度の差  $\Delta T'$  ( ${}^\circ\text{C}$ ) を  $10 \sim 50 {}^\circ\text{C}$  に規定したことを特徴とするものである。

【0036】塗膜の乱れを生じさせず十分な生産性を得るために、恒率乾燥時に乾燥風から塗膜に与える熱量  $Q$  を  $50 \sim 1000 \text{ kcal}/\text{m}^2 \cdot \text{hr}$  とすることに

より達成され、より好ましくは  $100 \sim 900 \text{ kcal}/\text{m}^2 \cdot \text{hr}$  である。

【0037】又  $h$  の値は  $5 \sim 30 \text{ kcal}/\text{m}^2 \cdot \text{hr} \cdot {}^\circ\text{C}$  とすることが好ましく、 $5 \sim 20 \text{ kcal}/\text{m}^2 \cdot \text{hr} \cdot {}^\circ\text{C}$  とすることがより好ましい。更に  $\Delta T'$  の値は  $10 \sim 50 {}^\circ\text{C}$  とすることが好ましく、 $10 \sim 40 {}^\circ\text{C}$  とすることがより好ましい。

【0038】本発明は溶媒中に溶質を溶解又は固体物を分散させたものを支持体上に塗布(塗工)、乾燥しうるものであれば溶媒種、溶質種、固体物種は限定されるものではなく、写真感光材料に代表されるゼラチン水溶液を塗工するものに対して高分子バインダーを溶解塗工する場合、溶媒は有機溶剤を用いることが多い。このような形態をとるものは磁性材料の塗工やPS版の感光層の塗工など数多くあるが、本発明の様な粘度範囲、塗布膜厚範囲にあるものとしては熱現像感剤が挙げられる。

【0039】以下に本発明を図を元に説明する。図1は乾燥ボックス内で搬送されながら支持体上に形成された塗膜が乾燥される様子を示した概略断面図である。

【0040】塗工から乾燥は一般的に次のよう経過をたどる。元巻から巻出された支持体3はダイコーター5に送られ塗膜を塗布される。支持体3は塗布面又は裏面、或いは両面をウェブクリーナーにより塗工直前に清掃することが好ましい。塗布面に異物が付着したまま塗布液を塗布すると塗膜中に異物が入り求める機能が得られなくなる他、塗布における筋故障の原因となることもある。バックロールを有する塗布形態で裏面に異物が付着したまま塗布すると異物により塗布時に支持体が持ち上げられ、局所的に求める塗布膜厚を得られなくなることがある。ウェブクリーナーとしては粘着ロールやクリーンなエアの吹きつけなどが用いられる。

【0041】塗布手段は本発明では限定されるものではなく、ダブルロールコーティヤリバースロールコーティヤなどのロールコーティヤやグラビアコーティヤ、コンマコーティヤ、エクストルージョンダイコーティヤなどが挙げられるが、本発明のように比較的厚膜の塗膜を形成する場合は塗工後の塗膜が重力により支持体上を流動してしまうことを防止するために、塗工直後の支持体の搬送方向が重力方向からより水平に近いことが好ましい。ダイコーターなどを用いてバックロールが存在する場合は両者の位置関係により塗工直後の塗膜が垂直、又はそれ以上に塗膜が重力方向に向く場合もあるが、その後次工程への支持体搬送角は水平に近いことが好ましい。

【0042】乾燥には赤外線やマイクロ波を用いたものもあるが、一般的には加熱、調湿した空気を塗膜に均一に吹き付けることにより塗膜から溶媒を蒸発させる手段が多く用いられる。又、蒸発した溶媒を効率よく補修し、又乾燥環境を制御するために乾燥工程はボックス状に作られる。この時乾燥ボックス1は一つでも良いが、複数の乾燥ゾーン2に区切ることにより各ゾーンの乾燥

条件を選択的に変更することができる。塗膜に空気を吹き付けて乾燥させる場合、各ゾーンの乾燥条件としては乾燥温度、吹き付ける空気の速度、吹き付ける空気の吹き出し口と塗膜の距離、新鮮空気量の供給量などが挙げられる。乾燥ゾーンへの空気の供給は塗膜へ吹き付けるためにノズル4（又はスリット）などからの空気の他に、塗膜へ空気の流れの影響を及ぼしにくい別の供給口を併用することもある。特に本発明で問題としているモトルムラが発生しやすい条件では塗膜へ吹き付ける空気の量が多いと塗膜を乱しやすいので、乾燥ボックス1内への十分な新鮮空気の供給のために別の供給口を設けることは重要である。塗膜の溶媒として有機溶剤を使用する場合は乾燥ボックス1内の溶媒蒸気濃度を監視することが一般的に行われる。乾燥ボックス1が複数の乾燥ゾーン2に分かれている場合には、各ゾーン毎に監視を行う。ドライヤー内で支持体3を裏面から支持ロールにより支えながら搬送する場合、支持ロールの進直度は10μm以下とすることが一般的であり、50μm以下とすることが好ましい。ダイコーター5から乾燥ボックス1内までは塗膜が未乾燥であり、表面に付着した異物は塗膜と接着してしまうことから周辺のクリーン度は高く保つ必要がある。乾燥ボックス1内のクリーン度は製造する製品の性格にもよるがクラス1000以下、好ましくはクラス100以下とすることが好ましい。又、乾燥ボックス1周辺では発塵源を遠ざける必要があり特に乾燥ボックス1内に空気を供給するためのファンや、震動源は乾燥ボックス1から隔離する必要がある。

【0043】塗膜内の残留溶媒量が仕上がった製品の機能に影響を与える場合には制御する必要があり、通常乾燥ボックス1を通過後、熱をかけるためのヒートキュアゾーンを設ける。乾燥ボックス1とヒートキュアゾーンの間は加熱した状態で連続的に繋がっていても良いし、一度常温又は必要に応じて冷却しても良い。ヒートキュアゾーン内の支持体の搬送は複数のロールにより行うこともできるし、エアフロート方式で支持体を浮上させて搬送しても良い。ヒートキュアゾーンは通常長い搬送系\*

\* 路を持つため、前後に支持体搬送のためのドライプロールや支持体搬送位置を制御するためのウェブガイドシステムを設けることが望ましい。

#### 【0044】

【実施例】以下、実施例を挙げて本発明を詳細に説明するが、本発明の態様はこれに限定されるものではない。

#### 【0045】塗布試料の作製

メチルエチルケトン（MEK）に固体分濃度25質量%で高分子バインダーを溶解させ、25°Cで剪断速度が0.1 s<sup>-1</sup>において粘度150 mPa·sの塗布液を得た。この塗布液をダイコーターにより100μmのPET支持体上にウェット膜厚100μmとなるよう塗布速度50m/minで均一に塗工した。

【0046】ダイコーターより塗工されたこの支持体を図1の様に3つの乾燥ゾーンからなる乾燥ボックスへ搬送し、スリットより加熱、調湿された空気を塗膜表面に吹き付けることにより乾燥して塗布試料を得た。得られた塗布試料について以下の評価を行った。

#### 【0047】（評価）

##### ・モトルムラ

発生状況は目視により観察評価したもので、10段階評価で1が悪く10が良い。

#### 【0048】実施例1

上記のようにして得られた塗布試料について、乾燥ボックスの3つの乾燥ゾーンの乾燥雰囲気温度を各ゾーンとも40°Cとし、乾燥風の強さを変化させて試料1及び2を得た。塗布後、所定時間後の試料は支持体の搬送を急停止させてサンプリングし、塗膜中の溶媒含有量（残留溶剂量）Dについて、試料中に残留するMEKをガスクロマトグラフィーにより分析した値を表1（試料1）及び表2（試料2）に示す。又、 $(\Delta D/D_0)/\Delta t$ についても同様に示した。尚、塗布直後（初期）の溶媒含有量D<sub>0</sub>は85g/m<sup>2</sup>であった。

#### 【0049】

##### 【表1】

塗布後の時間(sec)	5	10	15	20	25	30	35	40
D(g/m <sup>2</sup> )	78.6	70.2	58.6	46.5	33.9	22.1	10.6	0.01以下
(1-(D/D <sub>0</sub> ))×100(%)	8%	17%	31%	45%	60%	74%	88%	-
(ΔD/D <sub>0</sub> )/Δt(sec <sup>-1</sup> )	0.0151	0.0198	0.0273	0.0285	0.0296	0.0278	0.0271	-

【0050】

※※【表2】

塗布後の時間(sec)	5	10	15	20
D(g/m <sup>2</sup> )	74.8	44.3	14.2	0.01以下
(1-(D/D <sub>0</sub> ))×100(%)	12%	48%	83%	-
(ΔD/D <sub>0</sub> )/Δt(sec <sup>-1</sup> )	0.0240	0.0718	0.0708	-

【0051】 $(\Delta D/D_0)/\Delta t < 0.01 \text{ sec}^{-1}$  50 となった場合ドライヤー出口で未乾燥であったため実験

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中止となるなど、実用に適さなかった。一方モトルムラについて試料1は10点、試料2ではモトルムラは3点であった。

## 【0052】実施例2

上記のようにして得られた塗布試料を以下に示す表3の\*

試料	第1ゾーン	第2ゾーン	第3ゾーン	モトルムラレベル
3	40°C	60°C	80°C	10
4	20°C	30°C	40°C	プラッシング、未乾
5	40°C	50°C	60°C	未乾
6	60°C	70°C	80°C	6
7	40°C	70°C	90°C	8

【0054】表3から明らかなように、試料3は20～85°Cの間で20°Cの温度差を設け、段階的に乾燥温度を上昇させたためモトルムラの発生が抑えられ、良好であることが分かる。これに対し試料4、5の様に第1ゾーンが低温で、温度上昇が10°C差しかない場合は乾燥時間がかかりすぎ、乾燥が終了せず未乾となった。しかも試料4はプラッシングの発生も認められた。又、試料6の様に第1ゾーンを60°Cとし、温度上昇が10°C差の場合はモトルムラは改善されず、試料7の様に雰囲気温度が85°Cを越える場合もモトルムラは充分に良好とは言えない。

## 【0055】実施例3

上記のようにして得られた塗布試料について、乾燥ボッ※

\* 各乾燥条件にて乾燥させ、得られた試料のモトルムラを評価した。

## 【0053】

## 【表3】

※ クスの3つの乾燥ゾーンの乾燥雰囲気温度を各ゾーンとも40°Cとし、乾燥風の強さを変化させて試料8及び9を得、その塗膜の温度を非接触式の放射温度計にて測定した。測定は塗布後の所定時間後に通過する位置とそこから0.5秒後に通過する位置で測定し、2点の温度差と時間差(0.5秒)から $|\Delta T / \Delta t|$ を算出した。塗膜中の溶媒含有量(残留溶剤量)Dについて、試料中に残留するMEKをガスクロマトグラフィーにより分析した値を表4(試料8)及び表5(試料9)に示した。又、 $(\Delta D / D_0) / \Delta t$ についても同様に示した。尚、初期の溶媒含有量 $D_0$ は85g/m<sup>2</sup>であった。

## 【0056】

## 【表4】

塗布後の時間(sec)	5	10	15	20	25	30	35	40
D(g/m <sup>2</sup> )	78.6	70.2	58.6	46.5	33.9	22.1	10.6	0.01以下
$(1 - (D / D_0)) \times 100(\%)$	8%	17%	31%	45%	60%	74%	88%	—
$\Delta T / \Delta t (\text{°C/sec})$	6.1	2.9	1.8	1.2	0.9	0.7	0.6	—

## 【0057】

## ★ ★ 【表5】

塗布後の時間(sec)	5	10	15	20
D(g/m <sup>2</sup> )	74.8	44.3	14.2	0.01以下
$(1 - (D / D_0)) \times 100(\%)$	12%	48%	83%	—
$\Delta T / \Delta t (\text{°C/sec})$	8.6	4.9	1.4	—

【0058】表4及び表5から明らかなように、 $|\Delta T / \Delta t|$ が0.5～7°C/secの範囲にある試料8は優れておりモトルムラは10点、一方試料9のモトルムラは3点であった。

## 【0059】実施例4

ガラス転移点の異なる高分子バインダーに変更した他は☆

☆ 塗布試料の作製と同じ手順で試料10～13を作製した。この時の露点温度は13°Cであった。これを以下に示す表6の各乾燥条件にて乾燥させ、得られた試料のモトルムラを評価した。

## 【0060】

## 【表6】

試料	バインダー Tg(°C)	溶媒量1/2までの 乾燥雰囲気温度(°C)	溶媒量1/2以下での 乾燥雰囲気温度(°C)	モトルムラ レベル
10	72	30	60	10
11	72	10	60	プラッシング
12	72	30	75	3
13	85	30	80	4

【0061】表6から明らかなように、試料10の様に乾燥方法を溶媒量1/2までの乾燥雰囲気温度を露点温

度以上バインダーのガラス転移点Tg以下、又溶媒量1/2以下での乾燥雰囲気温度を先の乾燥雰囲気温度以上

バインダーのT<sub>g</sub>以下とすることでモトルムラの発生は抑えられ、良好な結果であった。一方試料11の様に溶媒量1/2までの乾燥雰囲気温度を露点温度以下とした場合ではブレッシングを起こし、又試料12の様に溶媒量1/2以下の乾燥雰囲気温度がバインダーのガラス転移点を越えた場合ではモトルムラは大きく悪化した。更に試料13の様に溶媒量1/2以下の乾燥雰囲気温度がMEK(79.6°C)の沸点を超えるとモトルムラは著しく悪化し、塗膜にガスが抜けたような気泡穴が発生した。

## 【0062】実施例5

\* 上記のようにして得られた塗布試料を以下に示す表7の各乾燥条件にて乾燥させ、得られた試料14~18のモトルムラを評価した。△T'は非接触式の放射温度計にて測定した塗膜温度と乾燥ボックス内温度の差とし、塗膜温度が乾燥に伴い変化するため幅を持つ。下記条件は恒率乾燥が終了するまでの条件であり、恒率乾燥の終点は塗膜表面に触れる乾燥していることで確認した。表中の乾燥時間は各試料で塗布後~乾燥終点までに要した時間を試料14で要した時間の何倍となるかで表した。

## 10 【0063】

\* 【表7】

試料	Q (kcal/m <sup>2</sup> ·hr)	h (kcal/m <sup>2</sup> ·hr·°C)	△T' (°C)	モトルムラ レベル	乾燥時間
14	340~440	20	17~22	10	1
15	680~940	40	17~24	9	0.6
16	60~240	30	2~9	10	1.7
17	34~40	2	17~20	10	9
18	1050~1230	30	35~41	3	0.4

【0064】表7から明らかなように、試料14では熱量Q、伝熱係数h、雰囲気温度と塗膜温度の差△T'の全てが本発明範囲の範囲内にありモトルムラが良好であった。試料15ではhが高いことからモトルムラに若干の低下が見られ、試料16では△T'が低いことから乾燥時間が若干長くなっている。試料17ではQ、h共に低いためモトルムラは良好であったが乾燥時間が長すぎ生産性が著しく悪化し、試料18ではQが大きすぎるためモトルムラは著しく悪化した。

## 【0065】

【発明の効果】本発明によれば、25°Cで剪断速度が0.1 sec<sup>-1</sup>において粘度が0.5 MPa·s~100 Pa·sの塗布液をウェット膜厚50 μm以上で塗布

※ した塗膜を乾燥する乾燥工程にて、塗布液を塗布して得た塗膜の乾燥速度を制御することによりモトルムラを抑制し、優れた平滑性を有する塗膜表面を形成することができるという顕著に優れた効果を奏する。

## 【図面の簡単な説明】

【図1】乾燥ボックス内で搬送されながら乾燥される支持体を表す概略断面図である。

## 【符号の説明】

- 1 乾燥ボックス
- 2 乾燥ゾーン
- 3 支持体
- 4 ノズル
- 5 ダイコーター

【図1】

